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RESEARCH TRIANGLE INSTITUTE

RTI/5676/93-Quarterly

October 1993

DEVELOPMENT OF LARGE AREA rf INDUCTION PLASMAS FOR COST EFFECTIVE DIAMOND

First Quarterly Report
July 20, 1993 - October 20, 1993

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Office of Naval Research
Program No.
N00014-94-C-0030

POST OFFICE BOX 12194 RESEARCH TRIANGLE PARK, NORTH CAROLINA 27709-2194

REPORT DOCUMENT PAGE

Form Approved
OMB No 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	January 31, 1994	Quarterly July 20,1993-Oct. 20,1993

4. TITLE AND SUBTITLE
Development of Large Area rf Induction Plasmas for Cost Effective Diamond

5. FUNDING NUMBERS
N00014-94-C-0030

6. AUTHOR(S)
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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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P. O. Box 12194
Research Triangle Park, NC 27709

8. PERFORMING ORGANIZATION REPORT NUMBER
83U-5676

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESSES(ES)

Office of Naval Research
800 N. Quincy Street
Arlington, VA 22217-5000

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; unlimited distribution

12b. DISTRIBUTION CODE

13. ABSTRACT
Work this quarter focused on initiation and testing of large area rf induction plasma system. The rf generator contained some parasitic oscillators which were suppressed. The plasma potential to ground was minimized. Diamond depositions from the large area tool have been documented by SEM, X-ray, and Raman. Diamond is being deposited over ~ 76% of the available substrate area. Gas stagnation is likely the problem limiting the deposition area to the 70% value. The program is directed at improving the gas distribution and pumping to eliminate the stagnation problem. Thick deposits are also being targeted next quarter to enable thermal conductivity testing.

14. SUBJECT TERMS
diamond, large-area, rf, induction

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	

NSN 7540-01-280-550
Form 298 (Rev. 2-89)

Standard

Prescribed

by ANSI Std Z39-18

TABLE OF CONTENTS

LIST OF FIGURES

1.0 INTRODUCTION

The program N00014-94-C-0030, "Development of Large Area rf Induction Plasmas for Cost Effective Diamond", began July 20, 1993. This document is the first quarterly report. The program seeks to significantly reduce the current costs of CVD diamond. Of all the competing thick-film diamond technologies (dc-arc jet, hot-filament, and microwave), the rf induction plasma technology has the potential to produce diamond at a low enough cost to compete with existing heat sinks ($\$10/in^2$). Currently, the rf plasma technology is in the proof-of-concept stage. Significant technical problems related to reactant gas distribution, plasma uniformity, substrate temperature control, and vacuum vessel design remain to be solved. However, there appear to be no "show stoppers", and substantial progress has been made this quarter in demonstrating large area diamond deposition from the rf induction plasma system:

1. equipment part count and equipment complexity have been reduced;
2. parasitics oscillations in the rf generator have been suppressed;
3. reactant gas feed has been placed under mass flow control;
4. plasma potential to ground has been minimized;
5. diamond has been deposited over 70% of the substrate area (900 cm^2);

Routine diamond growth from the large area rf induction plasma system is now possible. Diamond deposition proceeds everywhere in the system where the atomic hydrogen generated by the rf induction plasma is complemented by sufficient reactant feed and temperature.

2.0 EXPERIMENTAL RESULTS

Figures 1 - 4 show SEM and optical micrographs of diamond deposited in the rf planar reactor. The diamond is clearly faceted (111) material. Grain size in the deposited films increases with deposition thickness. 50 μm thick films are presently the thickest deposits. Those films are adherent to quartz substrates and do not show any cracking at this thickness. Renucleation does not appear to be dominating the growth process. Besides deposition on the quartz substrates, material has been recovered from other parts of the system. This material has been crushed into powders. The powders show clear faceting. X-ray analysis confirmed that the powders are diamond powder.

The primary problem with diamond deposition from the rf induction system this quarter has been a "hole" in the deposition pattern. Figure 5 shows a photograph of the deposition pattern. The photograph shows an annulus of diamond deposition near the periphery of the system. There exists near the center of the substrates a "hole" in which no diamond was deposited. We speculate that the hole is a consequence of gas stagnation and carbon depletion from the stagnated gas. Work next quarter will concentrate on eliminating this "hole" in the deposition pattern.

Besides materials deposition, RTI has been developing a downstream process monitor for the water-based process. A mass quadrupole is used to quantify the components of the output gas stream. We eventually intend to output data from the quadrupole to a computer program which will plot the C, O, and H atomic fractions on Bachmann's C-H-O phase diagram. For now, we have been monitoring the production of by-products, H_2, CO, and C_2H_2 as well as residual H_2O. We have observed that the ratio of $H_2O:C_2H_2:CO$ is critical to high quality diamond growth. Figures 6 and 7 show SEM micrographs of two samples grown under nearly identical conditions except for the $H_2O:C_2H_2:CO$ ratio. The sample grown with a high C_2H_2 background is small grain material. The sample grown with a lower C_2H_2 background shows excellent facetting and very little secondary nucleation. This work is ongoing on a separate reactor at RTI.

The reactor is undergoing a modification to provide for alternative reactant distribution and pumping. The new configuration will promote greater flexibility to support experimental and simulation efforts to optimize reactant distribution. In addition to the gas distribution change, the reactor will require increased pumping speed to handle increases in reactant throughput. This is particularly important to support studies of deposition rate and uniformity.

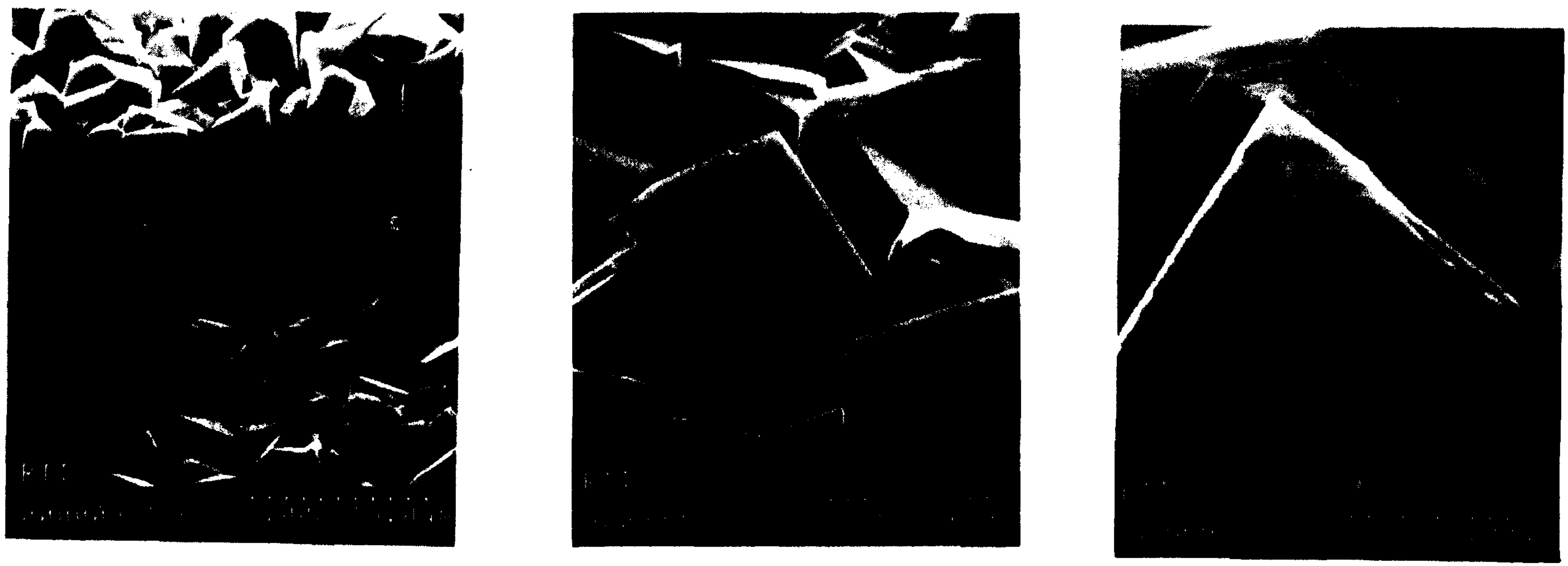

Figure 1

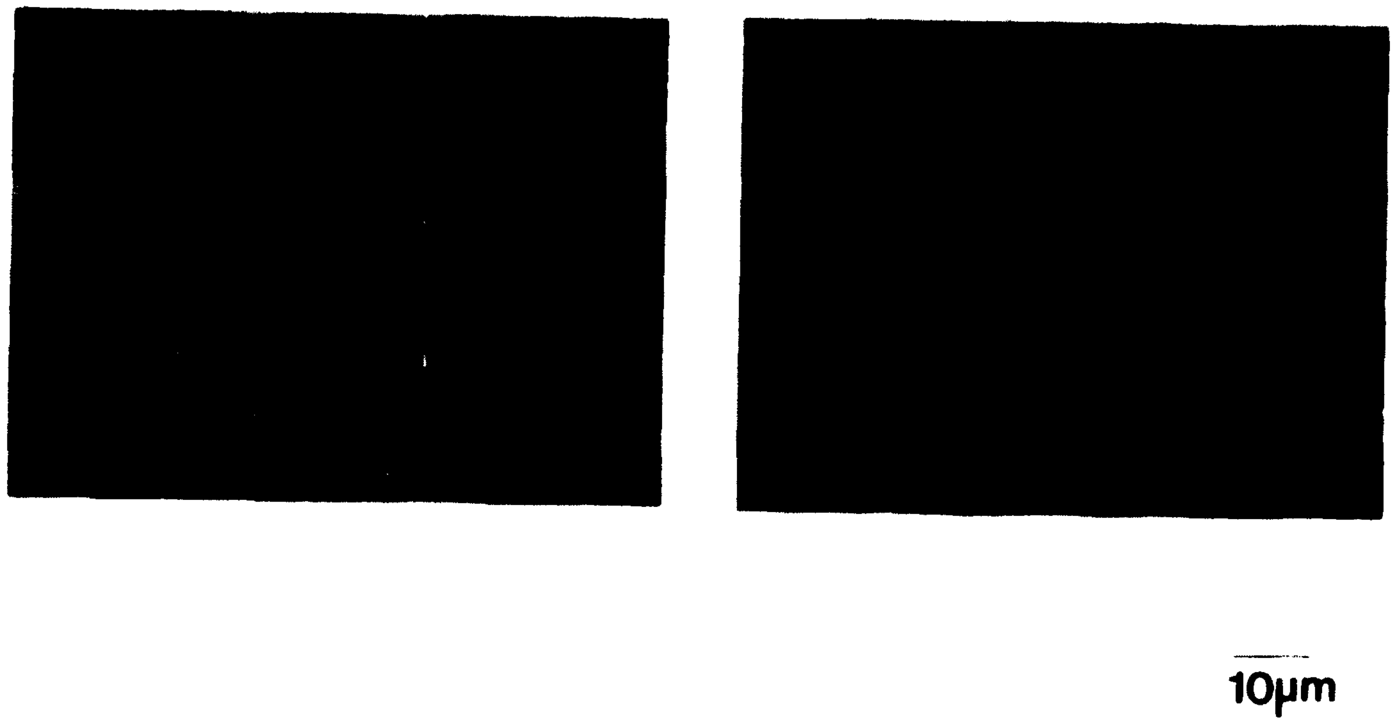

Figure 2

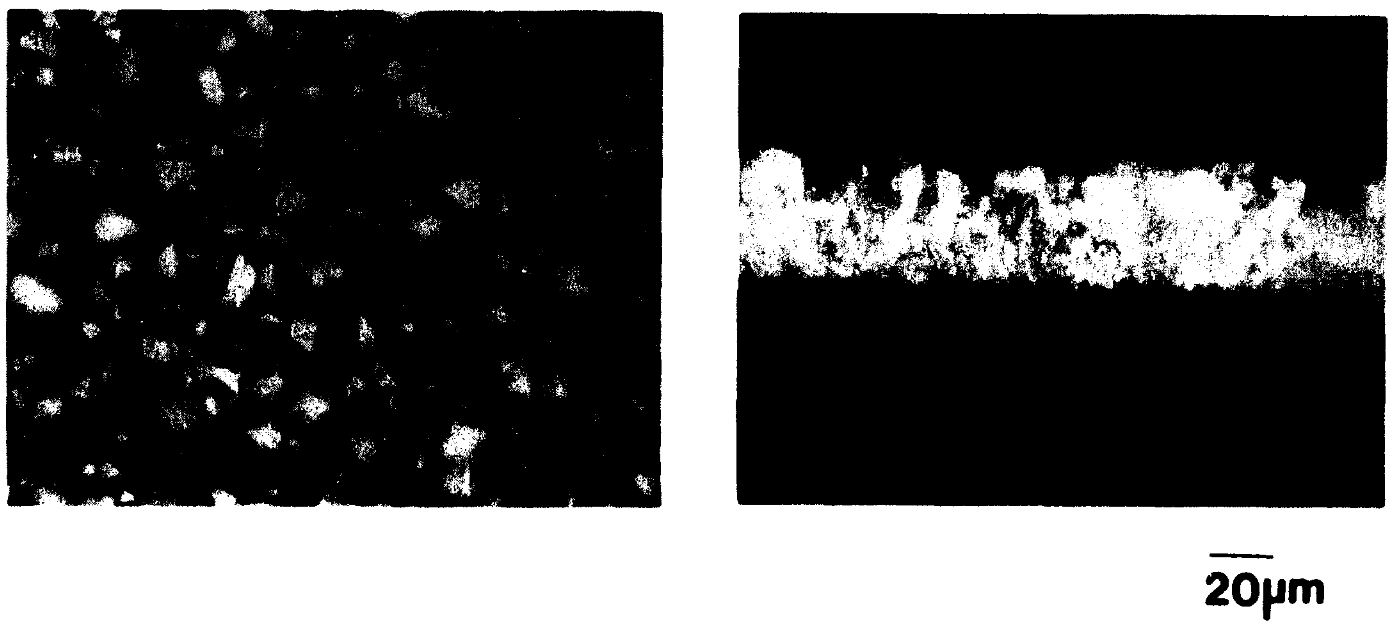

Figure 3

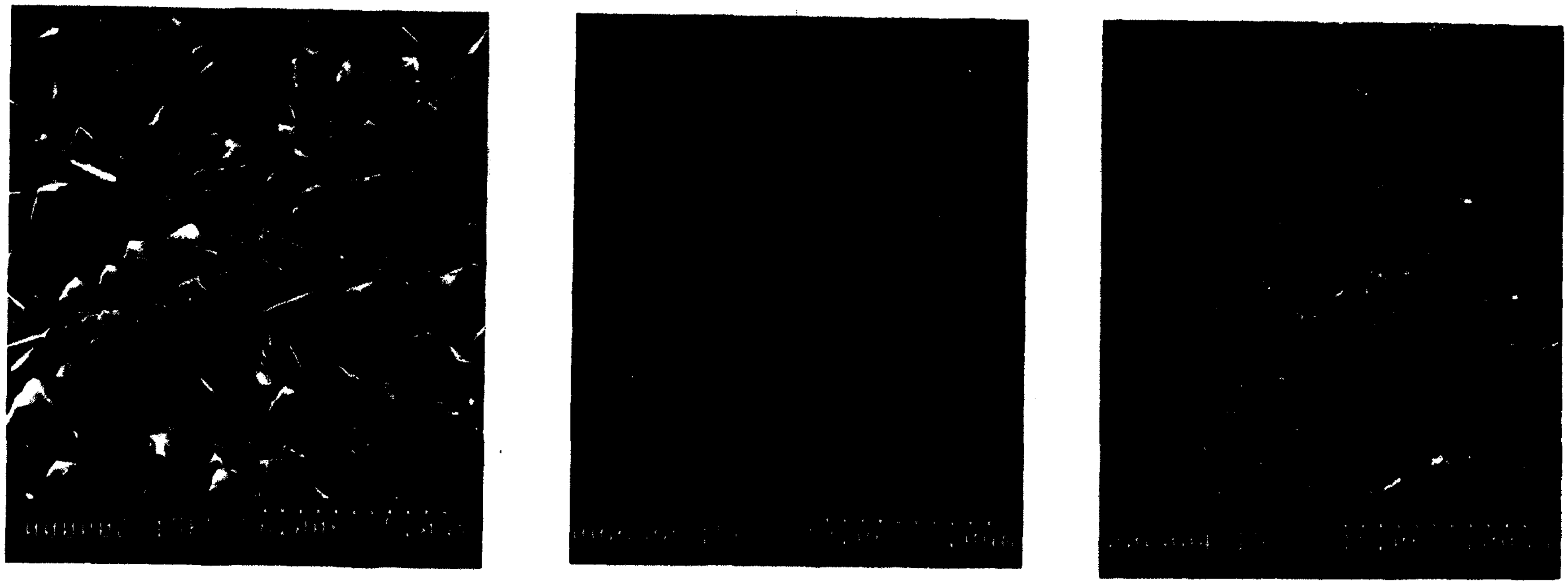

Figure 4

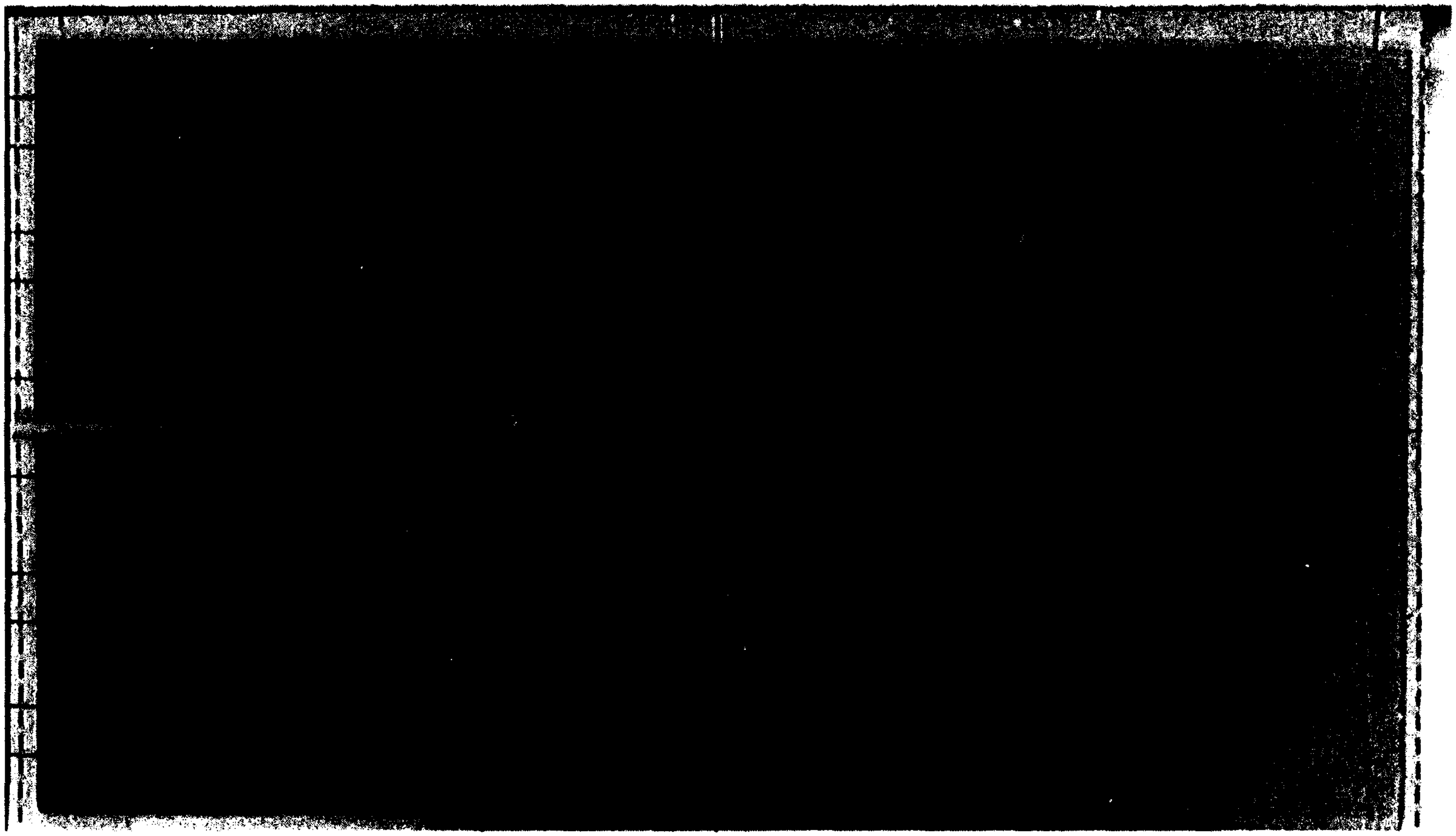

Figure 5:

$H_2O:CO:C_2H_2$ (26:33:7)

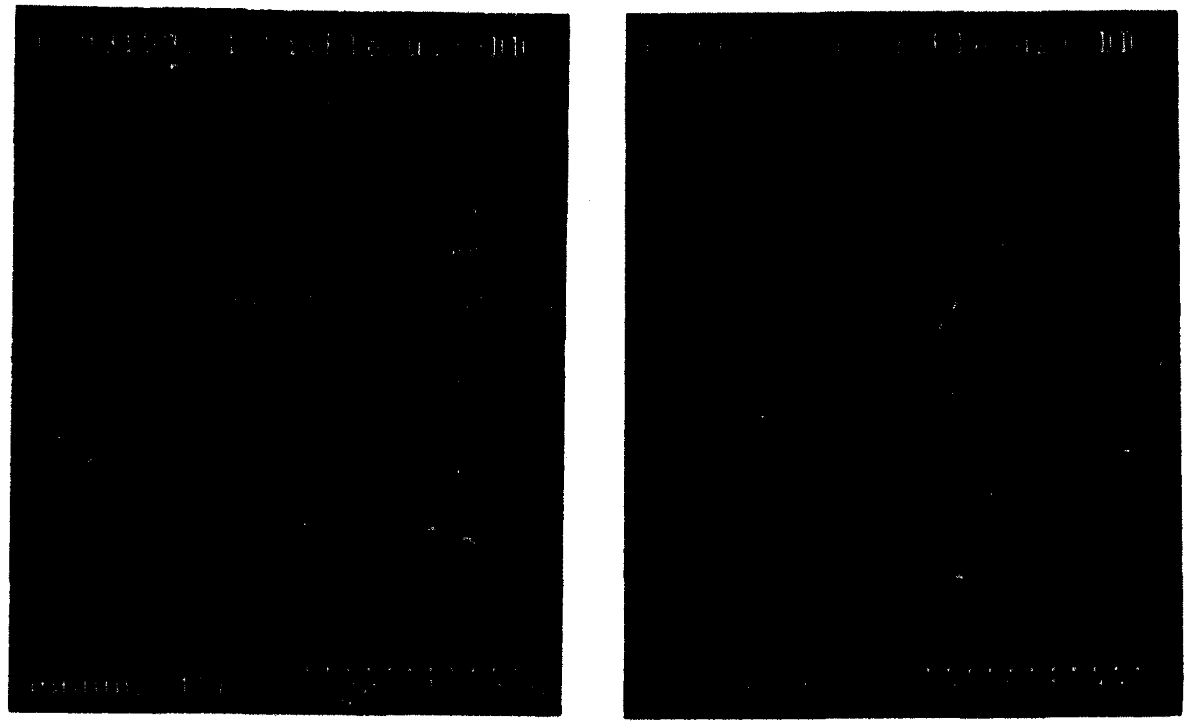

Figure 6

H_2O:CO:C_2H_2 (25:25:6)

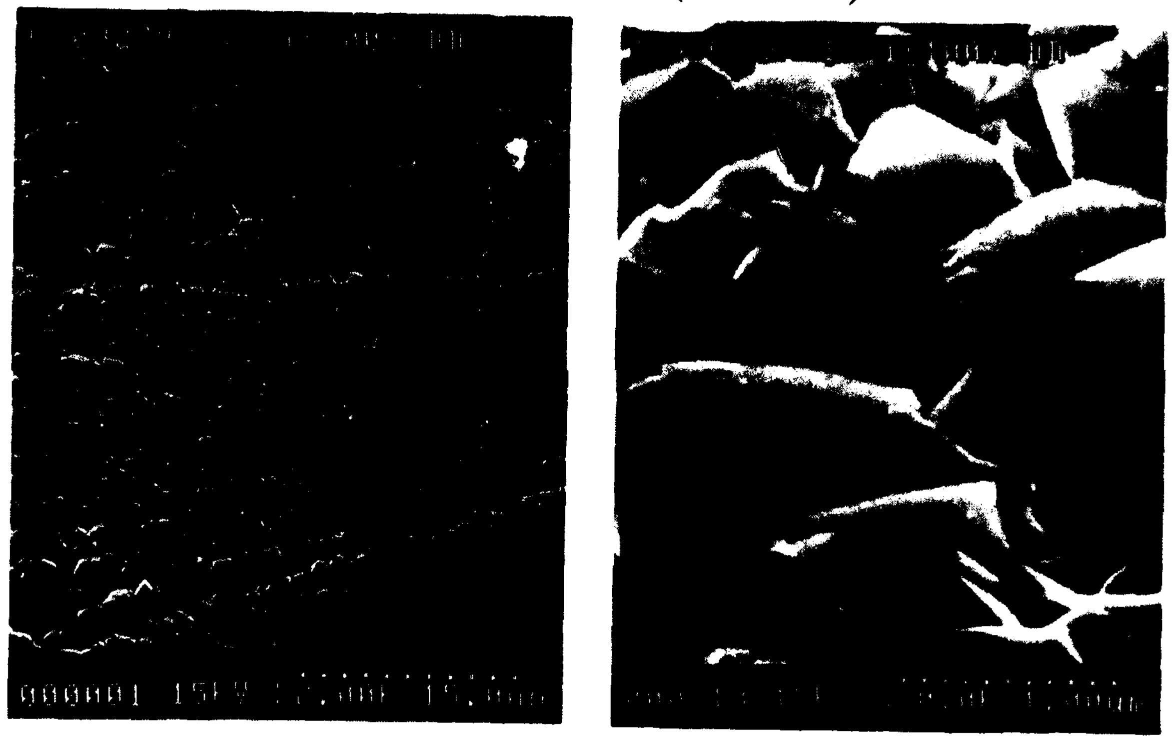

Figure 7

3.0 NEAR TERM TECHNICAL GOALS

Current directions of experimental thrust (under the ARPA/ONR program) are :

- complete modifications to gas delivery and exhaust system;
- fabricate 100 µm thick samples for thermal conductivity testing;
- explore higher growth rate conditions (higher power, pressure, flow rates);
- upgrade pumping to enable higher mass flow rates;